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Which is the Right Reference Surface Compression Value for Heat Treated Glass?

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The paper correlates the data recorded in Stazione Sperimentale del Vetro, in many years. The aim is to define a correlation between the following parameters in heat treated glass: 1) bending strength tested according EN 1288-3 ; 2) fragmentation tested according relevant Standards [EN 1863-1 2004, EN12150-1 2015, EN 14179-1 2016]; 3) surface compression stress measured with laser Gasp [ASTM C1279 2013]. For heat strengthened glass the fragmentation correlation due to the different crack path ("island" fragments instead of small fragments) is only related to conformity: Y/N. The research is the development of the previous ones [Schiavonato et al. 2005, Mognato et al. 2011] carried out at Stazione Sperimentale Vetro, increasing the experimental data (up to 2017)considering in detail the emissivity of coated glass and extending also to enamelled glass. The correlation between surface compression stress and mechanical strength and fragmentation is relevant for the manufacturer, who may use surface pre-stress measurement as a means of product control.

Keywords: Surface Compression, Bending Strength, Fragmentation, Thermally Treated

1. General

Thermally treated glass is used in many applications and the range of glass products is quite wide considering coated and enamelled glass.

Coated glass needs to be applied to respect energetic requirements: low-e, selective and reflective glass in function of the climatic zone and law mandatory values.

Enamelled glass is used for specific applications in which the designer would like to hide some elements or for artistic propose. The enamelled treatment could be applied uniformly on the whole surface or in some areas. The paint is applied and dried on glass surface; then the pane is thermally treated. The interaction between glass surface and paint is a tricky aspect due to the tensile stress that the frit induces at the interface and by the effect of pigment granules (Hreglich 2008); both weak the surface of application. This aspect is taken in account by Standards which reduce the minimum characteristic values for the mechanical strength (EN 1863-1 2004, EN 12150-1 2015, EN 14179-1 2016). In Italy a new Standard was published at the beginning of 2017 (UNI 11666 2017).

SSV carries out many experimental tests on all these products. The data are collected to evaluate a correlation between the Surface Compression Stress (SC) and the other characteristics: Fragmentation (FR) and Flexural Bending Strength (FB). This database started in 2002 and it is still going on. The data reported in the present paper had been collected until the end of 2017 and had been organised for: surface compression stress tested according (ASTM C1279 2013) / bending strength tested according (EN 1288-3 2000) / fragmentation tested according relevant standard (EN 1863-1 2004, EN 12150-1 2015, EN 14179-1 2016).

The aim of this paper is to evaluate and extend the considerations carried out in the previous papers (Schiavonato et al. 2005, Mognato et al. 2011, Redner et al. 2005) to coated and enamelled glass. Furthermore heat strengthened glass data were considered, whereas fragmentation is considered in terms of conformity Y/N (EN 1863-1 2004) due to the different crack path ("island" fragments instead of small fragments).

The correlation between surface compression stress and mechanical strength and fragmentation is relevant for the manufacturer, who may use surface pre-stress measurement as a means of product control.

1.1. Thermal process on heat treated glass

The soda lime silicate glass HS (compliant to EN 1863-1 2004) or TT (compliant to EN 12150-1 2015, EN 14179-1 2016) is a glass in which was induced permanent surface compressive stress through a controlled process of heating and cooling in order to increase mechanical and thermal strength; for TT product, additionally, the fragmentation characteristic limits the damage to people and/or things in case of breakage.

The heat transfer in the tempering process takes place through:

- Radiation (resistors in heating)
- Conduction (contact with the rollers)
- Convection (important in the case of coated glass)

The convention plays a crucial role in case of low-e glass: glass with high emissivity absorbs heat while one with low emissivity reflects it. The presence of faces with different emissivity may involve an asymmetrical heating resulting in curvature of the pane, with unlikely no homogeneous residual stresses.

After heating, in the first instants of air blowing, the glass surface is cooled more quickly than the centre of glass pane and in few seconds the temperature difference between the surface and the core of the pane reaches the maximum value. The quenching step is obtained by forced cooling whose time and intensity depend on the glass thickness.

Inadequate or inhomogeneous level of residual stress may be caused mainly by non- uniform heating of pane in its plane and/or between the two surfaces, with different quenching speed in the pane.

To avoid these problems a process control (FPC) which includes product control is mandatory.

1.2. Measurement of residual stress in heat treated glass as part of FPC

The measure of residual stress is carried on by photoelastic measurement, which has been widely developed in the recent years. Nowadays, the main instruments are: 1) GASP, registered trademark of Strainoptics Technologies; 2) SCALP, developed by GlasStress Ltd.

The measurement can be used to correlate SC with FB of FR values carried out by destructive tests. Redner wrote many papers on this topic (Redner 2003, Redner 2003, Feingold and Redner 2003, Redner and Hoffman 1997, Redner and Hoffman 2001, Redner and Bhat 1999) explaining the features of the GASP instrument and its capability to be used in FPC after glass tempering. Other authors proposed a new instrument (SCALP) based on scattered light polariscope technique (Anton et al. 2011, Aben et al. 2013) evincing that the residual stress in tempered glass can be highly inhomogeneous, both locally and globally.

1.3. Frame of the research

The research is developed according to test procedure reported in:

- EN 12150-1 for thermally toughened glass, in the following named TT
- EN 14179-1 for heat soaked thermally toughened glass, included in TT
- EN 1863-1 for heat strengthened glass, in the following named HS

which prescribe fragmentation (FR) test and four point bending (FB) test (EN 1288-3:2000), after measurement of surface compression (SC) stress (ASTM C1279 2013).

The data collected for the tests and used were:

- SC: surface compressive stress considered as mean value of five measure for each specimen;
- FR: number of fragments obtained according to (EN 12150-1 2015, EN 14179-1 2016);
- FB: flexural strength calculated at collapse load, following the equation defined in (EN 1288-3:2000).

The SC is correlated to FR and FB respectively.

Up to day, the ASTM C1048:2012 (ASTM C1048 2012) and ISO Standards (ISO/DIS 22509 2016, ISO/FDIS 12540 2016) specify a surface compressive stress requirement as showed in table 1; whereas the EN Standards define only the bending strength limits and the minimum number of fragments as reported in table 2.

Table 1: Reference Value of Surface Compressive Stress

Standard Reference	Heat Strengthened	Thermally Toughened
EN 1863-1:2012	No value is indicated	--
EN 12150-1	--	No value is indicated
EN 14179-1:2016	--	No value is indicated
ASTM C1048:2012	24÷52 MPa (thickness equal or lower than 6 mm)	69 MPa
ISO/DIS 22509 rev.:2016	25÷55 MPa	--
ISO/FDIS 12540:2016	--	80 MPa minimum for FB 90 MPa minimum for FR

Table 2: Minimum value of Bending Strength and number of fragments

Standard Reference	Float and coated	Enamelled
EN 1863-1:2012 (HS)	70 N/mm^2 (FB)	45 N/mm^2 (FB)
EN 12150-1:2015 (TT)	120 N/mm^2 (FB)	75 N/mm^2 (FB)
EN 14179-1:2016 (TT)	120 N/mm^2 (FB)	75 N/mm^2 (FB)
Glass thickness 4÷12 mm 15÷19 mm	40 TT (FR) 30 TT (FR)	40 TT (FR) 30 TT (FR)

The assessment for FR differs between HS and TT glass because the crack path is different. Therefore in case of HS glass the only indication of Conformity (C) or not (NC) has been considered to evaluate the SC necessary to comply. In case of TT glass the number of particles have been considered according the count procedure of Annex C (EN 12150-1 2015).

All the specimens were grouped as reported in tables 3a, 3b and 4a, 4b, where the number of available tested specimens are reported for the two correlations.

Table 3a: Number of TT tested for FR

TT-Thickness (mm)	4		5		6		8		10		12		15		Total	
	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC
Float	263	27 9%	267	19 7%	303	15 5%	306	19 6%	320	55 15%	237	38 14%	105	14 12%	1801	187 9%
B1: ε=0.89	30	0	20	0	95	0	65	0	30	0	--	--	--	--	240	0
B1_bis: 0.25<ε<0.89	20	0	5	0	53	7 12%	56	9 14%	60	5 8%	--	-	--	--	194	21 10%
B2: 0.1<ε<=0.25	37	3 8%	10	0	55	0	30	0	15	0	10	0	--	--	157	3 2%
B3: ε<=0.1	103	12 10%	25	0	102	13 11%	146	14 9%	90	10 10%	13	2 13%	--	--	479	51 10%
Enamelled	35	0	19	0	20	0	7	3 30%	25	0	5	0	--	--	111	3 3%

Table 3b: Number of HS tested for FR

HS-Thickness (mm)	4		5		6		8		10		12		15		Total	
	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC
Float	55	3 5%	88	3 3%	154	6 4%	149	21 12%	116	26 18%	50	10 17%	--	--	612	69 10%
B1: ε=0.89	--	--	10	0	10	15 60%	5	5 50%	5	0	5	0	--	--	35	20 26%
B1_bis: 0.25<ε<0.89	--	--	--	--	10	0	5	0	10	5 33%	5	0	--	--	30	5 14%

B2: 0.1<ε<=0.25	5	0	5	0	35	0	10	0	5	0	0	5 100%	--	--	60	5 8%
B3: ε<=0.1	5	0	25	0	35	5 13%	25	5 17%	28	10 26%	0	5 100%	--	--	118	25 17%
Enamelled	5	0	10	0	8	0	8	0	5	10 67%	--	--	--	--	36	10 22%

Table 4a. Number of TT tested for FB

TT-Thickness (mm)	4		5		6		8		10		12		15		Total	
	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC
Float	127	0	149	1 0.7%	193	1 0.5%	170	2 1.2%	237	1 0.4%	160	2 1.2%	84	0	1120	7 0.6%
B1: ε=0.89	21	3 12.5%	15	0	82	0	66	0	20	0	--	--	--	--	204	3 1.4%
B1_bis: 0.25<ε<0.89	15	0	4	0	33	1 2.9%	40	0	52	0	--	--	--	--	144	1 0.7%
B2: 0.1<ε<=0.25	25	0	7	0	51	0	20	2 9.1%	11	0	6	2 25%	--	--	120	4 3.2%
B3: ε<=0.1	89	0	25	0	99	1 1.0%	138	4 2.8%	99	1 1.0%	23	5 17.9%	--	--	473	11 2.3%
Enamelled	79	5 6.0%	34	0	28	0	16	0	72	4 5.3%	7	0	--	--	236	9 3.7%

Table 4b. Number of HS tested for FB

HS-Thickness (mm)	4		5		6		8		10		12		15		Total	
	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC	C	NC
Float	23	0	61	0	106	0	91	0	98	0	41	2 4.7%	--	--	420	2 0.5%
B1: ε=0.89	--	--	7	0	8	0	5	0	3	0	3	0	--	--	26	0
B1_bis: 0.25<ε<0.89	--	--	--	--	8	0	4	0	8	0	4	0	--	--	24	0
B2: 0.1<ε<=0.25	2	0	2	0	26	0	2	0	2	0	--	--	--	--	34	0
B3: ε<=0.1	2	0	15	0	42	0	20	0	33	0	2	0	--	--	114	0
Enamelled	10	0	20	9 31%	--	--	10	0	53	0	--	--	--	--	93	9 8.8%

The FB specimens are lower because, if the sampling did not pass FR, the test was stopped. For this reason the NC specimens are also limited.

EN Standards define B1 as coated glass with $0.89 \geq \varepsilon > 0.25$. In this range a large wide of products exist and the heat treatment differs greatly from glass to glass. For this reason the authors divided in B1 ($\varepsilon = 0.89$) and B1_bis ($0.89 > \varepsilon > 0.25$), but also in this B1_bis the ε range is too large.

Data are representative of Italian and European thermally treated glass producers.

As data refers to different producers, it means the tempering process differs for ovens and their technology of heating, convention and quenching, as for tempering recipes related to glass thickness and type.

Another aspect, which influences the correlation, concerns the rollers influence on glass bending strength. It is well known the influence of "tin" and "air" side referred to float glass. In thermally treated glass sometime this effect is also amplified on side placed in contact with tempering rollers. The authors carried out specific tests to evaluated the roller effect (Tab. 5) on the bending strength.

Table 5. Data of float glass

			SC (MPa)		FB (N/mm^2)	
Producer	Glass Type	Tensile side	Mean	Dev. St.	Mean	Dev. St.
A	10 mm Clear Float TT	no roller	107.0	6.8	194.4	23.8
		roller	106.0	7.4	138.2	8.3
B	10 mm Clear Float TT	no roller	105.5	1.9	202.0	26.8
		roller	104.6	2.2	165.0	18.1
	10 mm Clear Float HS	no roller	43.7	2.4	129.8	11.9
		roller	43.8	0.9	81.7	10.1

In these two very extremely cases roller effect is clearly evident. The SC values are equal inside the same sampling but the bending strength differs between "roller" and "no roller" side, independently from the "air" or "tin" side. In general the decrement of bending strength is accompanied by a decrement of standard deviation: defects, introduced by the roller, reduce data dispersion. The correlations of this paper (see tables 9-11) will be also affected by this effect.

The enamelling process weakens the glass surface and this aspect is well known, whereby the Standards define lower value of characteristic bending strength for enamelled glass, as reported in table 2. The enamelling process reduces the bending strength and the value dispersion too.

2. Experimental results

2.1. Fragmentation vs Surface Compression

All the specimens data (from 4 mm to 15 mm glass thickness) (surface compressive stress and particles number) were considered and the values of SC to get the conformity was reported in table 6 in function of glass thickness and type for TT. The data are plotted in figure 1 (float glass), 2a, 2b, 3a, 3b (coated glass), and 4 (enamelled glass).

Table 6. Minimum value SC (MPa) vs conform FR for TT in SSV tested specimens

	4 mm	5 mm	6 mm	8 mm	10 mm	12 mm	15 mm
Float	106	110	106	110	111	116	81
B1: ε=0.89	75	88	81	83	81	--	--
B1_bis: 0.25<ε<0.89	94	--	95	86	94	--	--
B2: 0.1<ε<=0.25	86	--	81	87	92	104	--
B3: ε<=0.1	115	85	107	97	100	108	--
Enamelled	96	97	97	91	96	--	--

The authors proposed in previous papers lower safety limit value. These values are not fully confirmed by the increment of test data. They may be also revised considering the coated B1_bis, B2, B3 and enamelled glass. The reason could be that the SC is measured at tin side (not coated) and the SC should be not homogeneous along the glass thickness, giving NC fragmentation pattern despite high SC values. Any way that not means that only at this high level the samples are conforms but that there is a certain degree on uncertainty according the SCV values you will adopt (see Tab. 7).

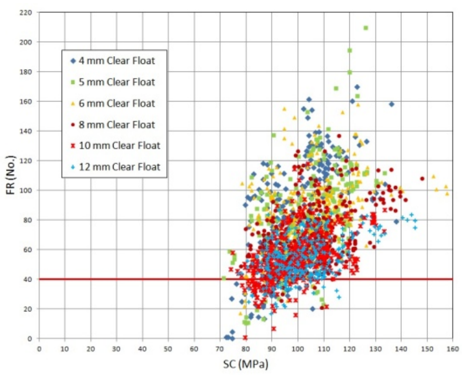
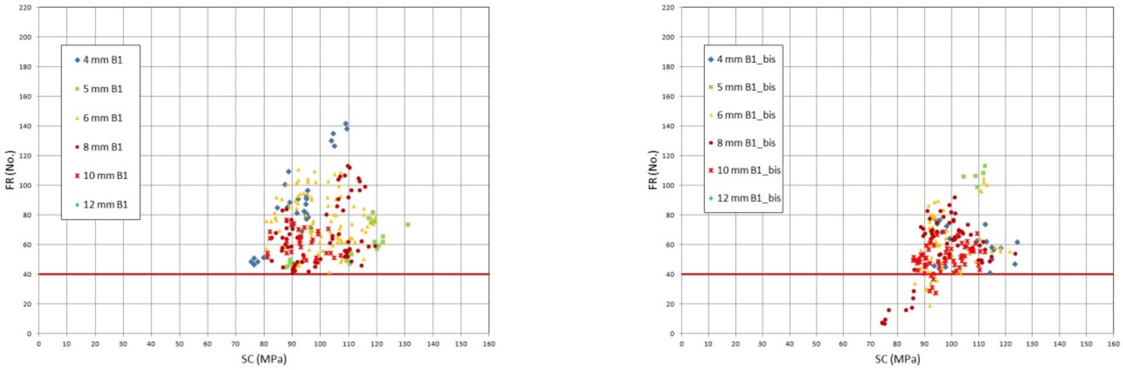
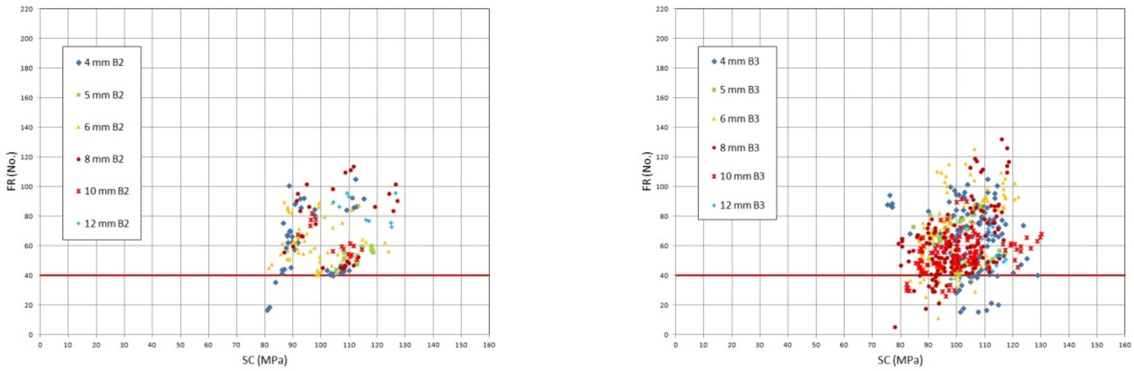


Fig 1. Correlation of surface compressive stress (SC) versus fragmentation (FR) for float glass



a) b)

Fig. 2 Correlation of surface compressive stress (SC) versus fragmentation (FR): a) for B1 coated glass; b) for B1_bis coated glass



a) b)

Fig. 3 Correlation of surface compressive stress (SC) versus fragmentation (FR): a) for B2 coated glass; b) for B3 coated glass

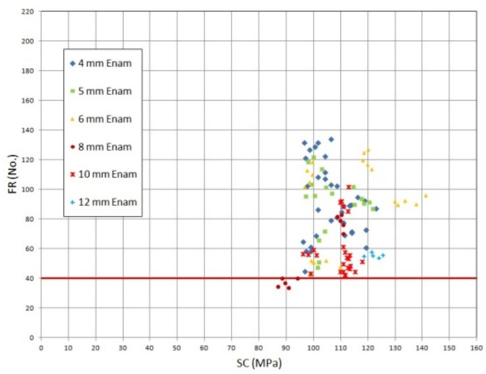


Fig. 4. Correlation of surface compressive stress (SC) versus fragmentation (FR) for enamelled glass

Table 7. Incidence value (%) and sample numbers () of NC data for SC (MPa) vs FR in TT with proposed SC value

	SC value	4 mm	5 mm	6 mm	8 mm	10 mm	12 mm	15 mm
Float	90/95	2(6)/2(5)	3(9)/2(5)	2(7)/1(4)	3(10)/1(4)	6(21)/3(12)	10(28)/5(15)	3(4)/3(4)
B1: ε=0.89	90	0	0	0	0	0	--	--
B1_bis: 0.25<ε<0.89	95	0	--	0	0	0	--	--
B2: 0.1<ε<=0.25	90	0	--	0	0	0	0	--
B3: ε<=0.1	95/100	10(12)/10(11)	0	5(6)/3(4)	1(1)/0	4(4)/0	13(2)/7(1)	--
Enamelled	95	0	0	0	0	0	--	--

In table 8 the collected data for HS are reported, considering conform and not specimens. In such case the SC limit is the higher one before get not conform breakage.

Table 8. Maximum value SC (MPa) vs conform FR for HS in SSV tested specimens

	4 mm	5 mm	6 mm	8 mm	10 mm	12 mm	15 mm
Float	67	61	62	48	43	47	--
B1: ε=0.89	--	67	51	56	63	--	--
B1_bis: 0.25<ε<0.89	--	--	53	45	45	--	--
B2: 0.1<ε<=0.25	62	63	63	55	51	--	--
B3: ε<=0.1	50	62	49	45	43	--	--
Enamelled	65	61	71	46	49	--	--

2.2. Flexural Bending Strength vs Surface Compression

Data of specimens with SC and FB measurement were considered. All the glass thickness and side in tension were considered (tin, air, coated, un-coated, enamelled) although the SC is measured only at "tin" side, "un-coated" and "un-enamelled" side. Moreover the data were not segregated, considering specimens with both central and edge fracture origin. They are reported in table 9 for thermally toughened and table 10 for heat strengthened glass, in function of glass thickness and type.

Table 9. Incidence value (%) and sample numbers () of NC data for SC (MPa) vs FB in TT with the proposed SC value

	SC value	4 mm	5 mm	6 mm	8 mm	10 mm	12 mm	15 mm
Float	90/95	0	0	0.3(1)/0.3(1)	0.8(2)/0.4(1)	0	1(2)/1(2)	0
B1: ε=0.89	90	0	0	0	0	0	--	--
B1_bis: 0.25<ε<0.89	95	0	--	2(1)/2(1)	0	0	--	--
B2: 0.1<ε<=0.25	95	0	--	0	0	0	--	--
B3: ε<=0.1	95	0	0	0	1(2)	0	17*(5)	--
Enamelled	95	0	0	0	0	1(1)	--	--

Note: * Sampling with high SC but with "roller effect"

In diagrams of figure 5, 6a, 6b, 7a, 7b and 8 the testing value are plotted, showing clearly the type of glass that were tested: heat strengthened and thermally toughened safety glass.

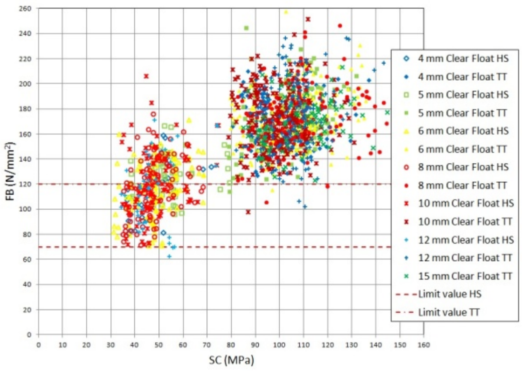
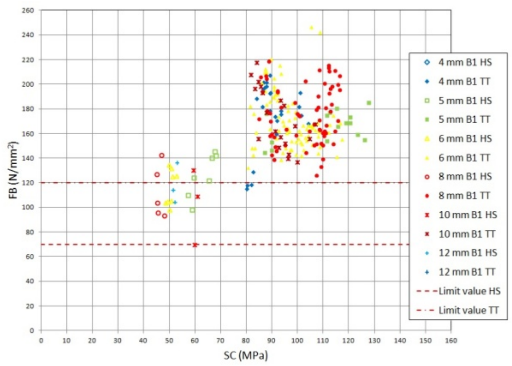
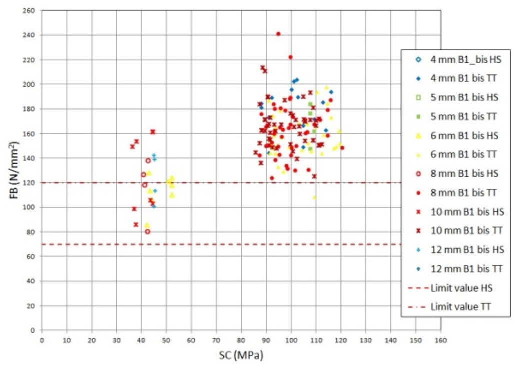


Fig. 5. Correlation of surface compressive stress (SC) versus flexural bending (FB) for float glass.

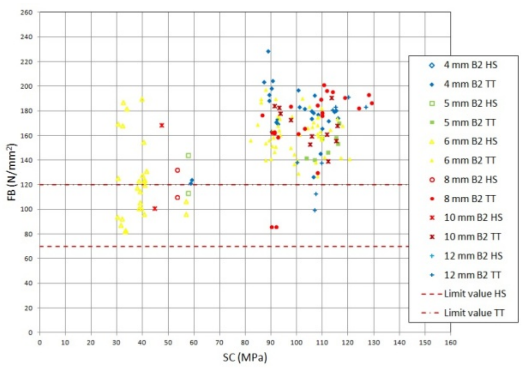


a)

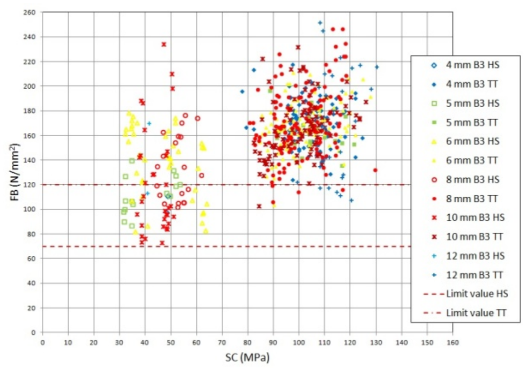


b)

Fig. 6 Correlation of surface compressive stress (SC) versus flexural bending (FB): a) for B1 coated glass; b) for B1_bis coated glass



a)



b)

Fig. 7 Correlation of surface compressive stress (SC) versus flexural bending (FB): a) for B2 coated glass; b) for B3 coated glass.

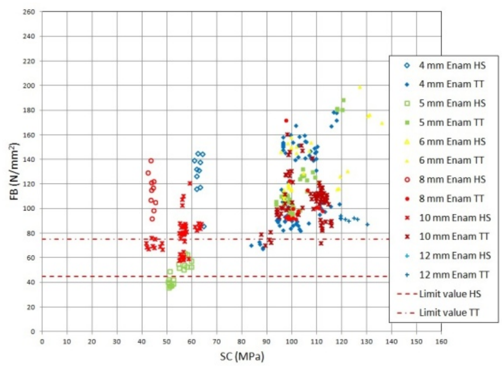


Fig. 8. Correlation of surface compressive stress (SC) versus flexural bending (FB) for enamelled glass.

The limit value of SC that has to be reached to respect the characteristic strength value of thermally toughened (TT) safety glass can be fixed 90 MPa for float and B1 glass and 95 MPa for other coated glass and enamelled glass (Tab. 9), having in such way a very low incidence of NC values.

In the case of heat strengthened glass (HS), the SC value of 35 MPa for float glass could be confirmed.A precise limit cannot be given due to the limited test samples for coated and enamelled glasses..

Table 10. Minimum SC values (MPa) vs conform FB for HS in SSV tested specimens

	4 mm	5 mm	6 mm	8 mm	10 mm	12 mm	15 mm
Float	37	37	31	35	34	34	--
B1: ε=0.89	--	57	48	--	--	--	--
B1_bis: 0.25<ε<0.89	--	--	42	--	36	--	--
B2: 0.1<ε<=0.25	59	--	30	--	--	--	--
B3: ε<=0.1	48	31	32	44	37	--	--
Enamelled	62	55	--	--	42	--	--

3. Conclusions

The elaborated data goes across many years of laboratory tests on different type of glass (heat strengthened and thermally toughened safety, coated and uncoated as enamelled) provided by different producers in Italy and in Europe.

The correlations between SC and FR or FB is accepted at Standard level (see ISO Standard) and is used during FPC (Factory Production Control) to evaluate the quality of process by a non destructive procedure. This procedure was defined as the measurements of surface compressive stress, as prescribed by EN 12150-2 for thermally toughened safety glass, EN 14179-2 for HST glass and EN 1863-2 for heat strengthened glass. The values were correlated to fragmentation (for TT) and to flexural strength (for HS and TT).

The limit value of SC proposed by the authors based on their experimental data are reported in table 11.

Table 11. Suggested SC value (MPa) respect FR and FB according SSV testing on estimation

Glass Type	FR_TT	FB_TT	FR_HS	FB_HS
	Minimum	Minimum	Upper	Minimum
Float	90	90	60	35
B1: ε=0.89	90	90	60	--
B1_bis: 0.25<ε<0.89	95	95	60	--
B2: 0.1<ε<=0.25	95	95	60	--
B3: ε<=0.1	95	95	60	--
Enamelled	95	95	60	--

Moreover for enamelled glass attention should be taken regards the frit effect, especially for HS.

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